(10) Japanese Patent Office (JP) (12) **Patent Gazette** (B2) (11) Patent Number: 3266995
(P3266995)
(45) Date Published: 18 March 2002 (24) Date Registered: 11 January 2002

(51) Int. Cl.[7] Identification No. FI
H 01 J 37/285 H 01 J 37/285
49/40 49/40

Number of Claims: 8 (15 pages total [in Japanese document][1])

(21) Application Number: H5-189610
(22) Date Filed: 30 July 1993
(65) Kokai (application publication) No.: H7-43373
(43) Date of application publication: 14 February 1995
Date Examination Requested: 26 June 1997

(73) Patentee: 000005108
Hitachi Manufacturing, Ltd.
4-6 Surugadai, Kanda, Chiyoda-ku, Tokyo

(72) Inventors: Yuuichi ISHIKAWA, Toshihiko YOSHIMURA, and Shinobu OHKIDO, all of
Hitachi Manufacturing, Ltd
Mechanical Research Laboratory
502 Kandatsu-machi, Tsuchiura-shi, Ibaraki-ken, Japan,
and
Osamu NISHIKAWA
Nishio Bldg., No. 608, 7-364 Kubo, Kanazawa-shi, Ishikawa-ken, Japan.

(56) References Cited: Japanese Patent Kokai (patent application publications):
S50-114969 (JP, A)
S61-263020 (JP, A)
H01-117234 (JP, A)
S62-232849 (JP, A)
S60-211756 (JP, A)

(58) Field of Search (Int.Cl[7], DB name):
H01J 37/285
H01J 49/40
H01J 9/02
H01J 9/42
H01J 37/06 – 37/077

Examiner: Shuuhei HORIBE

(54) Title of the Invention: Conductive Member Observation/Measurement Method and Apparatus Therefor

[1] Text enclosed in [square brackets] was added by the translator.

(57) **CLAIMS**

1. A conductive member observation/measurement method characterized in that

an extraction electrode supported in a mechanism capable of being moved in X-Y-Z directions is brought into the proximity of the forward surfaces of a plurality of tips (a multitip) formed on a conductive planar specimen;

a tip to be observed/measured is selected from the plurality of tips by the extraction electrode; then

imaging gas at a forward end of the selected tip is ionized by applying voltage to the selected tip in an imaging gas atmosphere, and the atomic arrangement at the forward end of the tip is analyzed/measured through the imaging gas ions.

2. A conductive member observation/measurement method characterized in that

an extraction electrode supported in a mechanism capable of being moved in X-Y-Z directions is brought into the proximity of the forward surfaces of a plurality of tips (a multitip) formed on a conductive planar specimen;

a tip to be observed/measured is selected from the plurality of tips by the extraction electrode; then

atoms are electrolytically evaporated[2] from the surface of the selected tip as positive ions, the electrolytically evaporated ions are detected by an ion detector, and the flight time of the ions from the selected tip to the ion detector is measured, for identifying the atoms.

[2] This is an obvious transcription error that is repeated several times. All instances of this term should read 'field evaporated.'

3. A conductive member measurement/observation method characterized in that

an extraction electrode supported in a mechanism capable of being moved in X-Y-Z directions is brought into the proximity of the forward surfaces of a plurality of tips (a multitip) formed on a conductive planar specimen;

a tip to be observed/measured is selected from the plurality of tips by the extraction electrode; then

a negative voltage is applied to the forward end of the selected tip, and the energy of electrons emitted from the tip is analyzed/measured.

4. A conductive member observation/measurement method characterized in that

an extraction electrode supported in a mechanism capable of being moved in X-Y-Z directions is brought into the proximity of the forward surfaces of a plurality of tips (a multitip) formed on a conductive planar specimen;

a tip to be observed/measured is selected from the plurality of tips by the extraction electrode;

after the forward end of the selected tip has been observed/measured, the extraction electrode is moved for observing/measuring a tip other than said selected tip.

5. A conductive member observation/measurement method as recited in any one of claims 1 through 3 characterized in that the ratio between the field strengths at the forward end of the selected tip and the forward end of the extraction electrode is at least 10:1, and the distance between the forward end of the selected tip and the forward end of the extraction electrode is no greater than 1 _m.

6. A conductive member observation/measurement apparatus comprising

a vacuum vessel;

an extraction electrode, supported in a mechanism that is capable of being moved in X-Y-Z directions, provided in a position opposite a plurality of tips (a multitip) formed on a conductive planar specimen,

a means of supplying imaging gas;

a means of applying voltage to a selected tip; and

a means of observing/measuring, through ions of the imaging gas, the atomic arrangement at the forward end of the selected tip.

7. A conductive member observation/measurement apparatus comprising

a vacuum vessel;

an extraction electrode, supported in a mechanism that is capable of being moved in X-Y-Z directions, provided in a position opposite a plurality of tips (a multitip) formed on a conductive planar specimen,

a means of applying voltage to a selected tip;

a means of effecting electrolytic evaporation of atoms from the surface of the selected tip, as positive ions;

a means of detecting said positive ions in an ion detector; and

a means of measuring the flight time of said positive ions from the selected tip to the ion detector.

8. A conductive member observation/measurement apparatus comprising

a vacuum vessel;

an extraction electrode, supported in a mechanism that is capable of being moved in X-Y-Z directions, provided in a position opposite a plurality of tips (a multitip) formed on a conductive planar specimen,

a means of applying voltage to a selected tip; and

a means of applying a negative voltage to the forward end of the selected tip, and analyzing/measuring the energy of electrons emitted from the selected tip.

DETAILED DESCRIPTION

0001

Industrial Field of Application

The present invention relates to apparatus for performing observation and measurement of conductive materials, and in particular to a method and apparatus having atomic-order resolution for analysis of the structure and composition of ultra-fine regions, and that is also effective for determining electron [energy] states. The invention also relates to a method for fabricating a specimen, the surface of which constitutes a beam source.

0002

Prior Art

In a conventional atom probe, high voltage is applied to a single needle-like specimen having a tip radius of no more than 0.1 μm. Surface atoms of the specimen are evaporated as ions by adding a pulsed voltage, or by irradiating the specimen with a pulsed laser. An evaporated atom is then identified by measuring its time of flight [from the specimen to a detector].

0003

The requirement that the radius at the tip of the needle be kept under 0.1 μm has to do with the voltage required to evaporate atoms. If the tip radius is too large, the voltage that must be applied to it [to effect field evaporation] becomes high enough to cause arcing. Also, even though a needle initially has a tip radius of less than 0.1 μm, as the evaporation process proceeds, the tip radius will gradually increase to the point where analysis can no longer be performed. For this reason, when performing analysis on the tip of a needle formed from linear-shaped specimen material [(wire, rod, etc.)], it was difficult to measure more than 50,000 atoms from one needle. This was a disadvantage in that it did not provide enough data to perform accurate statistical processing.

0004

Also, in the past, when performing measurements of adsorption, surface reaction, and boundary interfaces, it was necessary to take measurements from a large number of needles to verify the reliability of the data.

0005

More information on conventional technology of this type can be found in *Atom-probe Field Ion Microscopy,* by T. Tsong (1990, Cambridge University Press).

0006

Problems the Invention is to Solve

In conventional [atom probe] technology using a single needle, when the observation, analysis, and measurement of one needle was finished, [in order to perform additional] measurements, the needle/beam source had to be removed from the ultra-high vacuum, and replaced with a new one. This required an enormous amount of system down-time. Also, when a single needle was used as a specimen for atom probe or field ion microscopy (FIM), in many cases, certain data/statistical processing could not be performed [because there was not enough data]. Also, it was extremely difficult to fabricate needle specimens from two-dimensional samples having layered structures. There were also major limitations with respect to evaluation of boundary structures.

0007

Objectives of the present invention are to greatly reduce system down-time by using microtips as specimens and beam sources; to provide capability to analyze two-dimensional specimens; to provide a method and apparatus for analyzing atomic-order ultra-fine regions, with capability to perform measurements at a large number of points on a flat-geometry specimen without disturbing the ultra-high vacuum; and to provide a method for fabricating microtips appropriate for such analyses.

0008

Means of Solving the Problems

The above objectives are achieved using a scannable extraction microelectrode and a multitip. Firstly, for example, the objectives can be achieved by bringing an extraction electrode supported in a movable mechanism into the proximity of the forward surfaces of a multitip formed on a conductive planar specimen; selecting, with the extraction electrode, from the [tips of the] multitip, a tip to be observed/measured, then performing the desired observation/measurement of the selected tip.

0009

Secondly, the objectives can be achieved by moving an extraction electrode supported in a movable mechanism in order to observe/measure a tip other than a [previously] selected and observed/measured tip.

0010

Also, a conductive member observation/measurement apparatus according to the present invention can be realized by providing a vacuum vessel; an extraction electrode supported in a movable mechanism provided in a position opposite a multitip formed on a conductive planar specimen; and a means of observing/measuring the selected tip.

0011

0012

0013

0014

0015

Operation

Depending on the research objective, a planar specimen may be made from any one of a variety of materials such as metals, semiconductors, conductive glasses, conductive ceramics, and conductive polymers, by fashioning the material into sharp-ended microtips.

0016

A conical extraction electrode, supported in a mechanism that can be moved in the X-Y-Z directions, is brought closely adjacent to the front surface of one of the microtips of the specimen to select it for observation, measurement or analysis. After an imaging gas is introduced into the vacuum system, a high voltage (bias + pulse) is applied to the microtip [(to the specimen)]. This ionizes gas atoms at the tip of the needle, and the arrangement of atoms on the tip of the needle are observed through these ions.

0017

Continuing, surface atoms at the tip are evaporated as positive ions, which are caused to fly to an ion detector so that the atoms can be identified by measuring their flight times. Also, the apparatus can be operated as field emission microscope by applying a negative voltage to the specimen, wherein electrons are emitted from the needle tip so that electron states can also be investigated through measurement and analysis of the energy of the electrons.

0018

Once the observation and measurement of given tip is finished, the next tip is selected by the extraction electrode. This procedure continues until the desired analyses have been completed. In this manner, by

setting the specimen in the ultra-high vacuum system only once, it is possible to collect a large amount of data for analysis of surface and boundary structures and composition distributions, as well as the [energy] states of electrons.

0019

Embodiment

An embodiment of the present invention will now be described, with reference to the drawings. Fig. 1 shows a microtip specimen having a large number of tips **11** arranged on the flat surface of a common base **12**. The needle length and tip distribution interval [(spacing)] are selected according to the purpose for which the specimen is to be used.

0020

To fabricate this specimen, the tips are formed mechanically by using a cutter **22** to make lateral and longitudinal cuts in a flat sample **21**. This method can produce large numbers of tips in a short time, and also provides two-dimensional data for analysis. The apex angle **24** is determined by the blade angle **23** of the cutter.

0021

If the specimen is for FEM, FIM, atom probe, or position-sensitive atom probe (PoSAP) analysis, it is additionally immersed in electrolyte and electropolished to sharpen the apex to a curvature radius of no greater than 1000Å. In this regard, one advantage of the present invention is that a large number of sharp tips can be fabricated simultaneously in a short amount of time, whereas in the past, each individual tip had to be electropolished separately.

0022

In materials such as semiconductors and layered magnetic thin-films, the microtip specimen may be fabricated by a photolithography process, using ion milling, etc.. The microtip specimen can also be fabricated by positioning element clusters at set intervals on a flat sample as nuclei, and growing whisker crystals from these nuclei. In addition to the above uses, the present microtip specimen can be used as an electron beam source for FE-TEM (field-emission transmission electron microscopy) and electron beam holography.

0023

Next, a microtip for use as an atom probe FIM specimen will be described, with reference to Fig. 3. In place of a conventional single-tip specimen, a microtip **31** is installed in the ultra-high vacuum chamber of an atom probe FIM. The microtip is cooled to approximately 20 K by a cryostat **32**, and a positive high voltage (constant voltage) of a few kV is applied to the specimen.

0024

A tapered conical extraction electrode **33** having a diameter of less than 1 μm is placed opposite one of the tips **34**, and brought closely adjacent thereto. A positive high voltage of a few kV is also applied to this extraction electrode. In this embodiment, the extraction electrode **33** is formed by electrocasting or deformation processing, and is supported in a mechanism whereby it can be moved in the X, Y, and Z directions. Also, to prevent damage to the tips and base by high density field emission current from the extraction electrode, the shape of the extraction electrode is configured so that the ratio of field strength between the end of the tip and the end of the extraction electrode will be at least 10:1, and the electrode is brought to the optimum position closely proximate to the tip (no more than 1 μm away from the tip).

0025

In addition, atoms can be field-evaporated from the surface of the tip by superimposing, on the constant voltage, a pulsed voltage of 10 to 20 % of the constant voltage [(i.e., increasing the standing voltage by 10 to 20 % during the pulse time)]. Such field-evaporated atoms become ions **35**, which fly to a detector **36**. If a continuous string of voltage pulses is applied, surface atoms will be extracted, one at a time, from the end of the tip, thus making it possible to obtain a density profile of single-atom layers from the surface of the tip. If an inert gas such as He or Ne is introduced in the absence of such voltage pulses, an FIM image showing the two-dimensional arrangement of surface atoms can be observed on a screen **37**.

0026

After approximately 50,000 or more ions have been taken from the tip **34**, the radius of curvature at the end of the tip increases to where the voltage required for field evaporation is too high for analysis to continue. In addition, if a negative voltage is applied to the needle, the FIM can be operated as an FEM, in which electrons are emitted from the tip of the needle.

0027

A faraday cup **36** may be inserted in back of the probe hole at the center of the screen **37**, for analyzing electron energy. This will make it possible to determine the [energy] states of electrons in an area on the surface of the tip corresponding to the probe hole.

0028

In a conventional atom probe FIM system, analysis would end at this point. In the present invention, however, the extraction probe **33** is simply moved, and brought closely adjacent to the next tip **38**, and similar analysis performed on that tip. Thus by repeating the above analysis for each tip, a large amount of

data can be obtained from a single specimen, and density distribution in the planar direction of the specimen can be investigated.

0029

Thus we see an additional advantage of the invention in that the surface of the specimen may also be used as an electron/ion source capable of emitting large numbers of electrons or ions. Also, if the tip of the extraction probe can be refined to a diameter of no more than a few thousand angstroms, since it would then be in the same dimension as that of the flat specimen, and of the scan probes currently used in scanning tunneling microscopy (STM), and if atomic-order protrusions were formed on the specimen surface by the machining disclosed in this embodiment, but without making microtips, the surface itself could serve as an electron/ion source.

0030

Effects of the Invention

According to the present invention, a large number of microtips are arranged on a single specimen, for use as a specimen or a beam source. This eliminates the need to change out the specimen to analyze each tip, thus improving the operational ratio [(the ratio of operating time to down-time)]. In addition, this also makes it possible to perform a large number of measurements without disturbing the ultra-high vacuum.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 shows a microtip [specimen].

Fig. 2 is schematic drawing showing the fabrication of microtips by a mechanical cutting method.

Fig. 3 is a schematic drawing showing a method for using a microtip as a specimen for atom probe FIM analysis.

Reference Numbers

11: Tip	12: Base	21: Flat-plate specimen	22: Cutter	23: Blade angle
24: Tip angle	31: Microtip specimen		32: Cryostat	33: Extraction Electrode
34: Tip	35: Ion	36: Detector	37: Screen	38: Faraday cup

DRAWINGS

Fig. 1

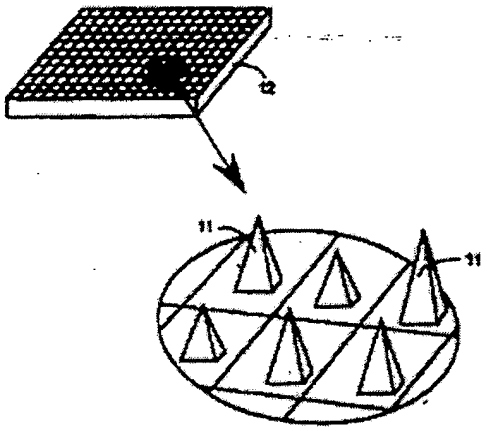

Fig. 2

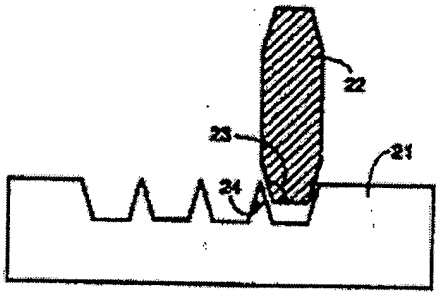

Fig. 3